

Diffusion Reaction Behaviors of U-Mo/Al Dispersion Fuel

Ho Jin Ryu, Jong Man Park, Chang Kyu Kim, Yeon Soo Kim, and Gerard L. Hofman

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The uranium (U)-molybdenum (Mo)/aluminum (Al) dispersion fuel that is currently under development for high-performance research reactors has shown complicated diffusion reaction behavior between the U-Mo particles and the Al matrix. Diffusion reactions in U-Mo/Al dispersion fuels were characterized by out-of-pile annealing tests and in-pile irradiation tests in the HANARO research reactor. The effect of the addition of a third element such as silicon (Si), Al, or zirconium (Zr) to U-Mo fuel, and the addition of Si to the Al matrix on the diffusion reaction were also investigated. The growth rate and activation energy for the reaction phases of U-Mo/Al dispersion fuels were obtained. The effect of alloying a small amount of a third element in U-Mo and of Si in the Al matrix on diffusion reaction kinetics was negligible in annealing tests conducted at ~550 °C. γ phase stability in the U-Mo alloy was enhanced by the addition of 0.1 to 0.2 wt.% Si. The Si accumulated in the interdiffusion layer of U-Mo/Al-Si dispersion fuel annealed at ~550 °C, whereas Zr migration to the interdiffusion layer of U-Mo-Zr/Al was negligible.

Keywords annealing tests, diffusion reaction, dispersion fuel, irradiation tests, reaction kinetics, reaction layer

1. Introduction

Uranium (U)-molybdenum (Mo)/aluminum (Al) dispersion fuel is being developed for high-performance research reactors under the Reduced Enrichment for Research and Test Reactors program.^[1] An issue that has become apparent in the course of the development of this fuel is a diffusion reaction between the fuel particles and the matrix.^[2] The diffusion reaction between U-Mo and the Al matrix adversely affects irradiation performance of the U-Mo/Al dispersion fuel by increasing fuel dimensions and by decreasing the thermal conductivity of the fuel.^[3] In addition, in irradiation at high temperatures to extended burnup, the reaction phases of U-Mo/Al dispersion fuels developed large pores. Therefore, it is necessary to understand the diffusion reaction behavior to mitigate the effect of U-Mo versus Al interdiffusion. This has motivated numerous irradiation and out-of-pile tests worldwide.

Three intermetallic phases, namely, UAl_2 , UAl_3 , and $UAl_{4,4}$, exist in the U-Al binary system.^[4,5] However, dif-

fusion reaction phases with the average composition ranging from $(U,Mo)Al_5$ to $(U,Mo)Al_7$ have been observed in U-Mo/Al irradiation tests,^[6] as well as in annealing tests of cast U-Mo-Al alloys^[7] and U-Mo versus Al diffusion couple tests.^[8] The reaction phases with higher Al content are considered to be less stable during the irradiation.^[9] As a means of preventing the formation of a reaction phase with higher Al content, the modification of U-Mo fuel with the addition of transition metal elements, such as zirconium (Zr) and titanium (Ti), and modification of the Al matrix with silicon (Si) addition have been proposed.^[9] This calls for characterization of the diffusion behaviors of alloying elements in U-Mo and Al matrix.

In this article, experimental results are presented for the diffusion reaction between U-Mo and the Al matrix of U-Mo/Al dispersion fuel both from annealing tests performed at high temperature and from irradiation tests. The results with the modified fuel and matrix are also included.

2. Experimental Procedures

Spherical U-Mo powder with a rapidly solidified γ (body-centered cubic [bcc]) U structure was fabricated by a centrifugal atomization method.^[10] Ternary U-Mo-X alloy powder was fabricated by adding 0.1 to 1.0 wt.% Si, Al, or Zr. The Mo content varied from 6 to 10 wt.%. The U-Mo-X powder and pure Al powder or Al-2 wt.% Si powder were mixed and hot-extruded at 400 °C with an extrusion ratio of 38:1 to make rod-shaped dispersion fuel samples. The volume fraction of U-Mo in dispersion fuels varies from 10 to 50 vol.%.

Annealing tests of U-Mo/Al dispersion fuel samples were carried out at 500 to 580 °C for 1 to 36 h in a vacuum-sealed quartz tube. Irradiation tests of 4 g-U/cm³ (25 vol.%) and 6 g-U/cm³ (38 vol.%) U-Mo/Al dispersion fuel were carried out in the HANARO reactor.^[11]

X-ray diffraction (XRD) techniques were used to compare the stability of the γ phase in U-Mo-X alloys. The microstructures of the U-Mo/Al dispersion fuel samples

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were characterized by scanning electron microscopy (SEM), and concentration profiles in reaction phases were obtained by electron probe microanalysis equipped with an energy-dispersive x-ray (EDX) spectrometer.

3. Results and Discussion

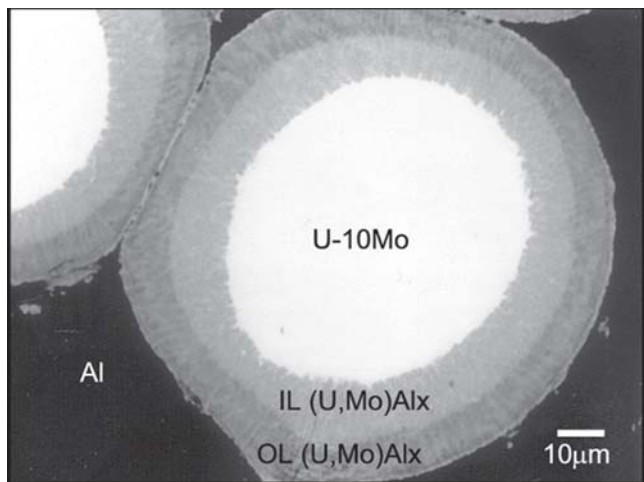
Figure 1 shows the micrographs of 10 vol.% U-10Mo/Al dispersion fuel after annealing at 550 °C for 9 h (Fig 1a) and 36 h (Fig 1b). The reaction phases, shown in gray, have formed between the spherical U-Mo particles (bright phase) and the Al matrix (dark phase). The reaction product shows three distinct layers. The composition of the layers was measured by EDX spectrometer. The two major layers are composed of a mixture of two phases—a feature that is common in ternary diffusion. The thin layer farthest outside could not be characterized in this study. Table 1 summarizes the average compositions of U, Mo, and Al in the two major

layers. The average composition of the inner layer (IL) is close to that of $(U,Mo)Al_3$, and the outer layer (OL) composition is similar to that of $(U,Mo)Al_{4.4}$. However, these

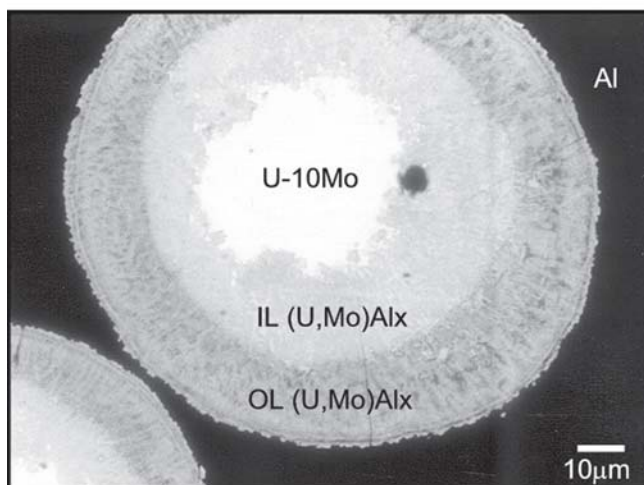
Table 1 Compositions of U, Mo, and Al in the reaction layers of 10 vol.% U-Mo/Al dispersion fuels annealed at 550 °C for 25 h

Element	550 °C/25 h	
	IL, at. %	OL, at. %
U	18.0	13.8
Mo	4.9	2.9
Al	77.1	83.3
Al/(U + Mo)	3.4	5.0

Note: IL, inner layer; OL, outer layer

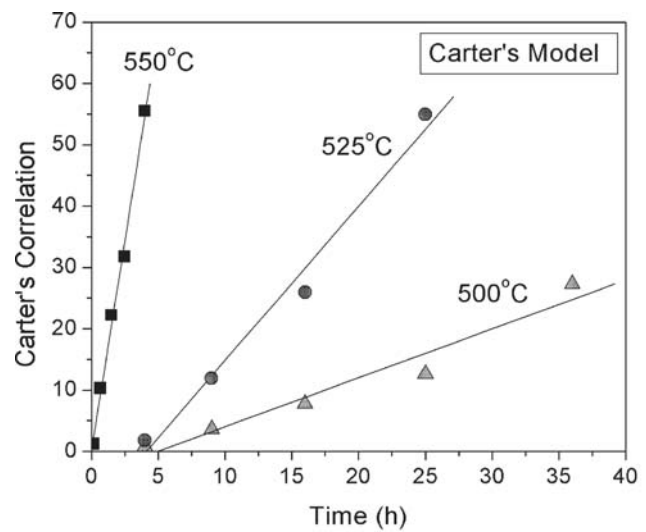


(a)

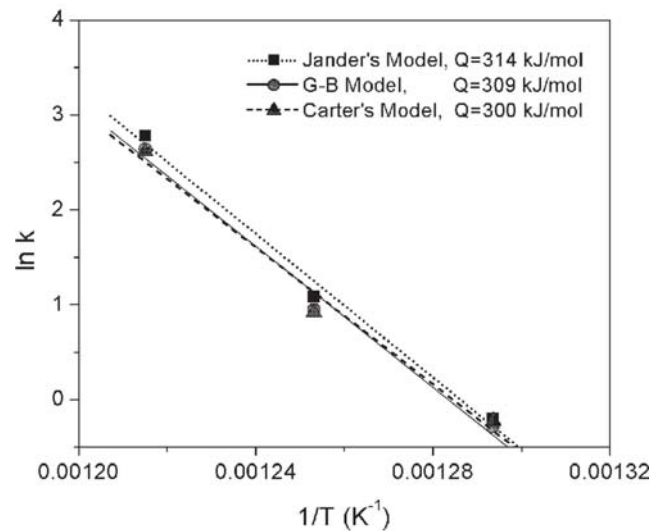


(b)

Fig. 1 SEM micrographs showing the reaction layers in 10 vol.% U-10Mo/Al dispersion fuels annealed at 550 °C (a) for 9 h and (b) for 36 h



(a)



(b)

Fig. 2 (a) Variation of the rate constant of the total reaction layer with annealing time at 500, 525, and 550 °C based on Carter's model. (b) Arrhenius plot used for the activation energy calculation according to three diffusion kinetics models

are average compositions of the finely dispersed multiphase layers.

The growth of the reaction product follows a parabolic rate law in the U/Al and U₃Si/Al systems based on Jander's model.^[12-14] To describe the reaction kinetics of spherical particles, the Ginstling-Brounshtein model was used.^[15] It takes the following form:

$$[1 - 2\alpha/3 - (1 - \alpha)^{2/3}] = kt/r_0^2 \quad (\text{Eq 1})$$

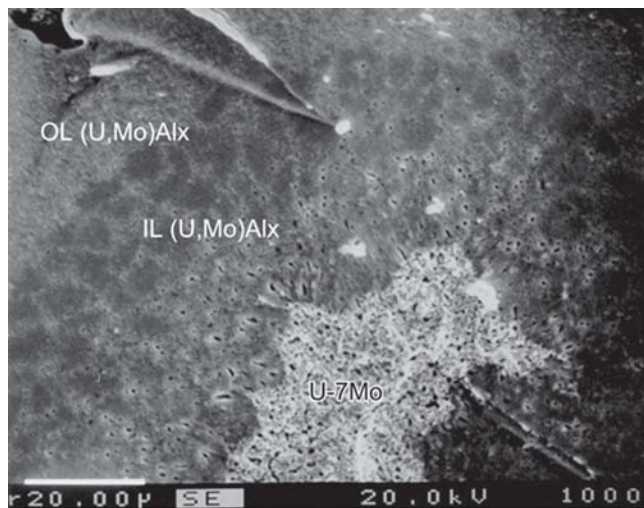
where α is the reacted volume fraction of the particle at time t , k is the reaction rate constant, and r_0 is the initial particle radius.

$$\alpha = 1 - \left(1 - \frac{x}{r_0}\right)^3 \quad (\text{Eq 2})$$

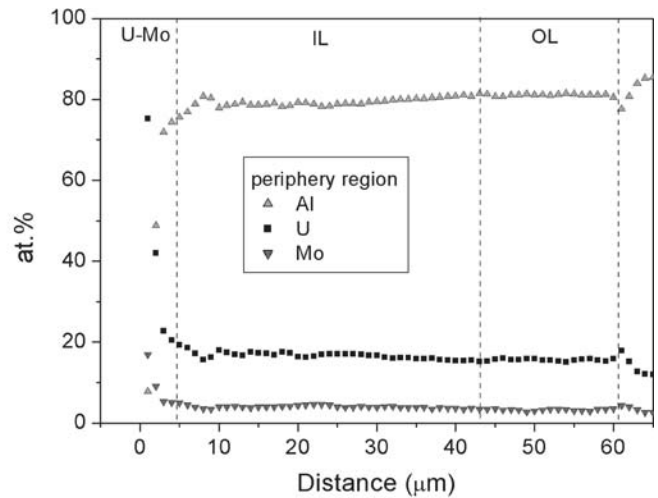
where x is the reaction layer thickness. In situations where the volume of the reaction product is different from the volume of the spherical particle consumed, Carter's model is more accurate.^[16]

$$[1 + (z - 1)\alpha]^{2/3} + (z - 1)(1 - \alpha)^{2/3} = z + 2(1 - z)kt/r_0^2 \quad (\text{Eq 3})$$

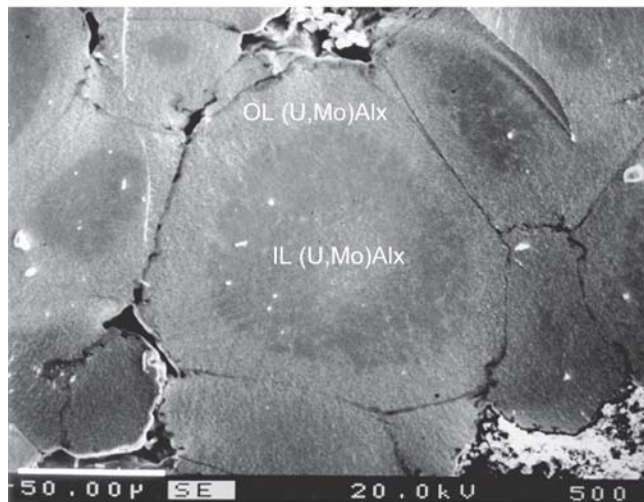
where z is the ratio of the volume of the reaction product to the volume of the spherical particle consumed. Based on the reaction rate constants obtained from time and temperature conditions in which (U,Mo)Al₃ is the dominant phase (shown in Fig. 2a is an example for Carter's model), the effective activation energies of the reaction process were calculated using an Arrhenius plot, as shown in Fig. 2(b). The activation energy values according to the three models were similar to one another (300–314 kJ/mole).



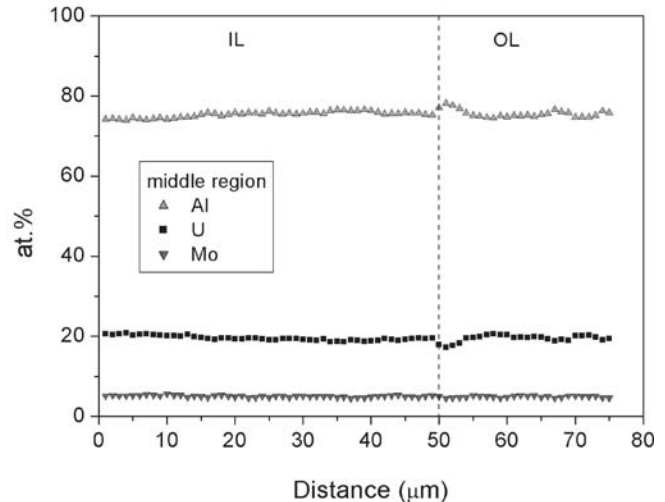
(a)



(b)



(c)



(d)

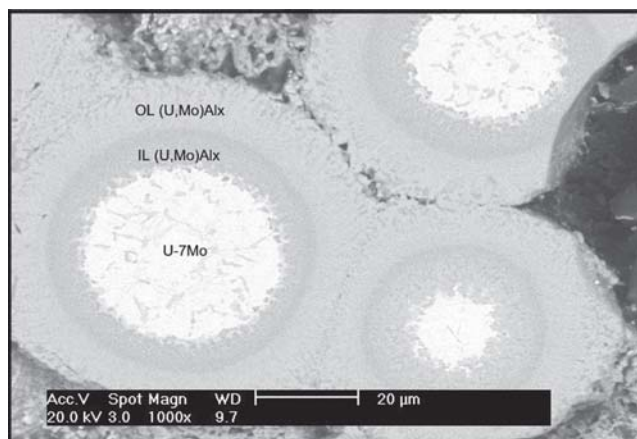
Fig. 3 SEM micrographs and corresponding composition profiles of 4.0 g-U/cm³ U-7Mo/Al dispersion fuel after 68 at.% U-235 burnup in the HANARO reactor. (a, b) Fuel rod, periphery region. (c, d) Fuel rod, middle region

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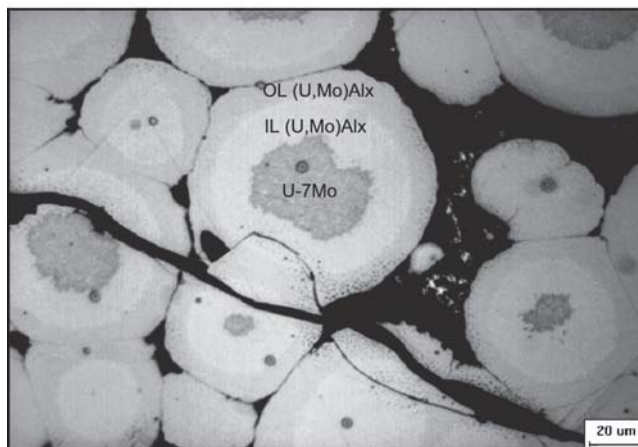
The microstructures of the reaction phases of the U/Mo/Al dispersion fuel irradiated in the HANARO reactor were observed, as shown in Fig. 3.^[17] Two distinctive layers were observed in the irradiated U-Mo/Al dispersion fuel. This multilayer formation of the irradiated U-Mo/Al dispersion fuel appears to be due to the thermal diffusion from the standpoint that low-temperature-irradiated fuel does not show the multilayered reaction phase.^[18] This is an interesting feature of our high-temperature irradiation tests, confirming the fact that at high temperatures the irradiation effect becomes negligible and thermal diffusion is more important. This is due to the recombination of defects and the annihilation of irradiation damage. Composition profiles for the 4 g-U/cm³ (25 vol.%) fuel with a burnup of 68 at.% U-235 depletion showed that the reaction product in the rod periphery had a composition of approximately (U,Mo)Al_{4.4}, as shown in Fig. 3(a), whereas that in the rod interior region had a composition of approximately (U,Mo)Al₃, as shown in Fig. 3(b). One possibility of this different composition of reaction product across the radial direction is the change in the boundary condition for the diffusion process due to the depletion of the Al matrix. On the other hand, when the Al matrix was still available, the Al concentration was found to be high, up to (U-Mo)Al₇ in other irradiation tests.^[6]

When 6 g-U/cm³ (38 vol.%) U-7Mo/Al dispersion fuel was annealed at 600 °C for 24 h, the Al matrix was completely consumed. Two distinctive layers in the reaction phase were observed, as shown in Fig. 4(a). The OL contained a low Mo content (~1.6 at.%), and the IL showed a high Mo content (~5.7 at.%), while the Al content of each layer remained almost the same as that listed in Table 2. Figure 4(b) shows a cross-section image of the dispersion fuel rod with 6 g-U/cm³ terminated at 8% burnup in the HANARO reactor irradiation test due to a very high fuel temperature.^[11] When the micrograph of this fuel was compared with the findings of an out-of-pile annealing test conducted at 600 °C for 24 h, multilayer formation and Al matrix depletion were commonly observed. When all of the Al matrix is consumed, which is frequently observed in high-temperature irradiation tests, the reaction phase turns into multilayers because the diffusion couple is no longer U-Mo versus Al. Instead it is U-Mo versus (U-Mo)Al_x. For this case, Al from (U-Mo)Al_x contacting U-Mo continues to diffuse into U-Mo, forming a reaction layer with lower Al composition at the interface. Therefore, the composition variation of the reaction phase is due to the availability of the Al matrix to the reaction phase. It appears that the overall microstructure of the interaction layers formed in the in-pile tests is similar to those observed in the out-of-reactor annealing test. This suggests that the fuel irradiation temperatures were high enough to be comparable to the 550 to 600 °C range in the annealing tests.

The modification of U-Mo fuel and Al matrix was accomplished by alloying a third element to U-Mo fuel and Si to Al matrix. According to ternary phase diagram, these elements, if present in the reaction product, suppress the formation of Al containing compound higher than (U,Mo)Al₃.^[9] For example, addition of more than 6 at.% Zr



(a)



(b)

Fig. 4 (a) SEM micrograph showing the depletion of the Al matrix and multilayered interaction phases in 6 g-U/cm³ U-7Mo/Al dispersion fuel annealed at 600 °C for 24 h. (b) SEM micrograph of U-7Mo/Al dispersion fuel irradiated to ~8 at.% U-235 at high temperatures showing the similar multilayered morphology as that in the annealing test in (a)

Table 2 Compositions of U, Mo, and Al in the reaction layers of 6 g-U/cm³ (38 vol.%) U-7Mo/Al dispersion fuel annealed at 600 °C for 24 h

Element	Composition	
	IL, at. %	OL, at. %
U	15.4	20.6
Mo	5.7	1.6
Al	78.9	77.8
Al/(U + Mo)	3.7	3.5

Note: IL, inner layer; OL, outer layer

was effective in increasing the stability of UAl₃ without transformation into UAl₄.^[19]

The stability of the γ (bcc U) phase in U-Mo alloys was compared with its stability in U-Mo-X ternary alloys by annealing at 500 °C before investigating the diffusion reaction behaviors of U-Mo-X/Al or U-Mo/Al-Si. When 1 wt.%

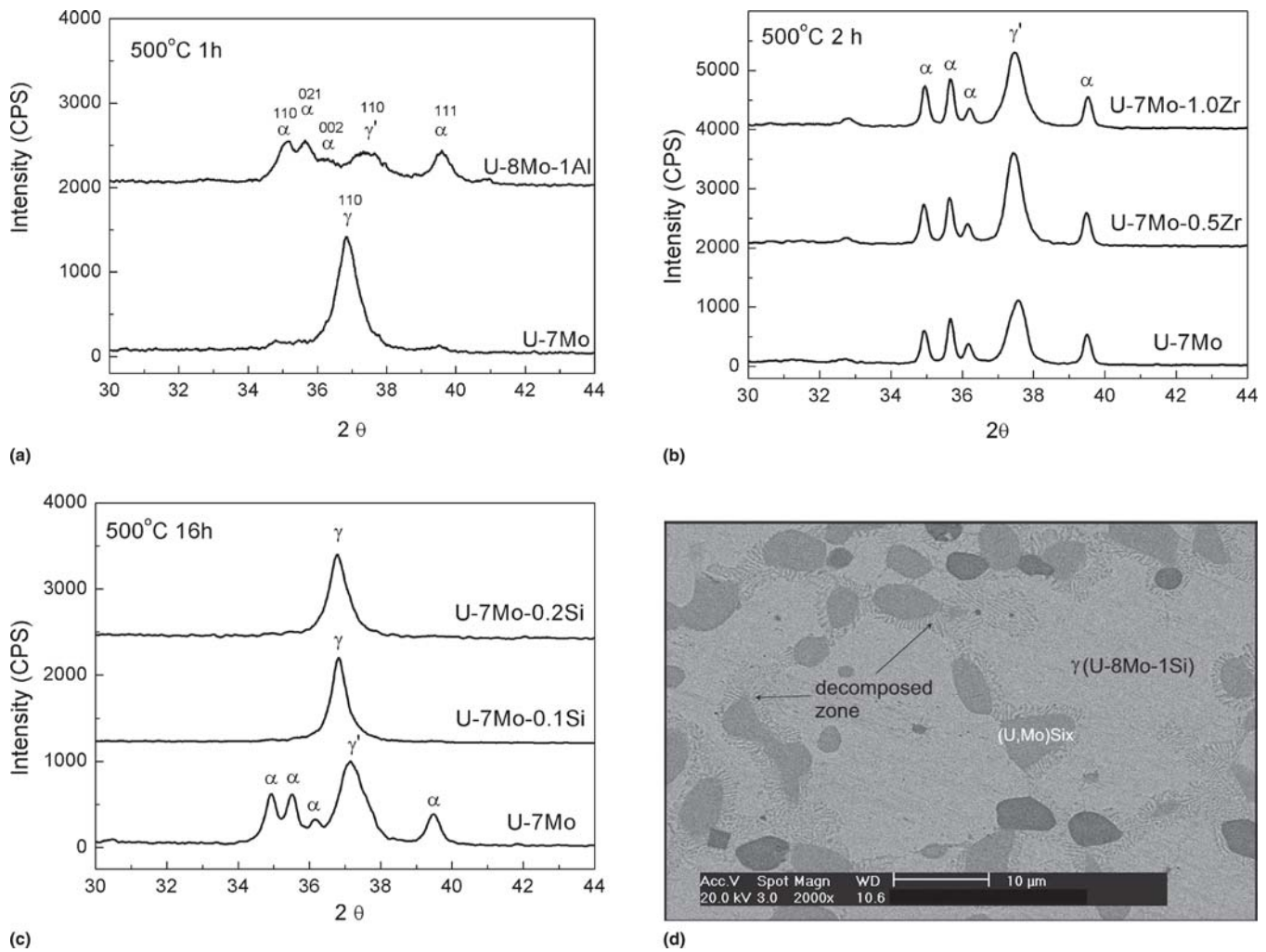


Fig. 5 XRD patterns showing the stability of the γ phase in the U-Mo-X alloys: (a) U-7Mo and U-8Mo-1Al annealed at 500 °C for 1 h; (b) U-7Mo and U-7Mo-Zr (0.5 and 1.0 wt.%, respectively) annealed at 500 °C for 2 h; and (c) U-7Mo and U-7Mo-Si (0.1 and 0.2 wt.%, respectively) annealed at 500 °C for 16 h. (d) SEM micrograph of U-8Mo-1Si annealed at 500 °C for 24 h

Al is added to a U-Mo alloy, the γ phase decomposed earlier than in the U-Mo alloy, as shown in Fig. 5(a). The γ -stabilized U-8Mo-1Al alloy was transformed into the two-phase mixture of the α (orthorhombic U) and γ' (tetragonal U_2Mo) phases after 1 h at 500 °C. U-7Mo and U-7Mo-1Zr showed similar decomposition behaviors, as shown in Fig. 5(b). Si, however, showed an excellent γ -stabilizing power, as shown in Fig. 5(c). Even after 16 h of annealing, the U-7Mo-(0.1 or 0.2 wt.%)Si alloy showed a typical diffraction pattern of a γ -stabilized U alloy when compared with U-7Mo. The SEM micrographs of the U-8Mo-1Si alloy after annealing are shown in Fig. 5(d). The γ phase surrounding the Si-rich precipitates, $(U,Mo)Si_x$, was transformed at the grain boundaries into a lamellar structure chiefly because the Si-rich precipitates have a high Mo content and an Mo-depleted zone was formed adjacent to the Si-rich precipitates. The behavior of Si-rich precipitates in the diffusion reaction between U-Mo and Al was observed using diffusion couple tests of U-8Mo-1Si and Al at 550 °C for 16 h. The $(U,Mo)Si_x$ precipitate phase in the U-8Mo-1Si

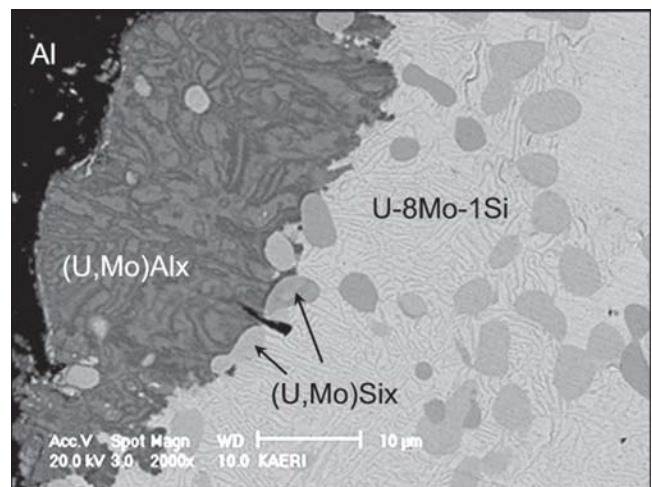
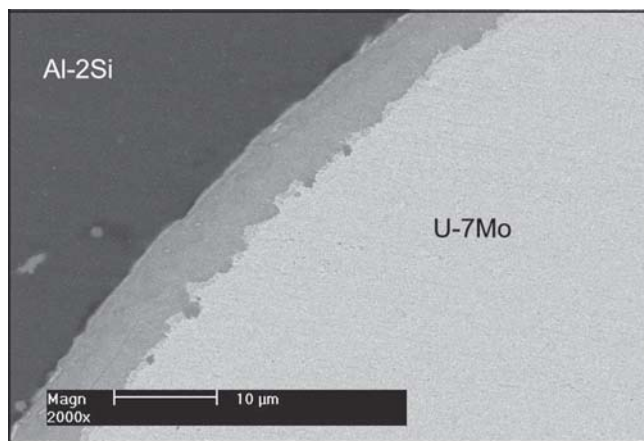
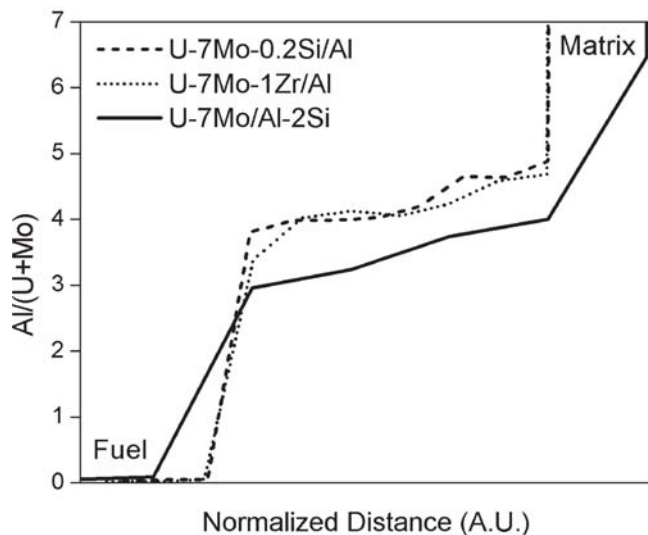


Fig. 6 SEM micrograph of diffusion reaction phases in a U-8Mo-1Si and Al diffusion couple annealed at 550 °C for 16 h



(a)

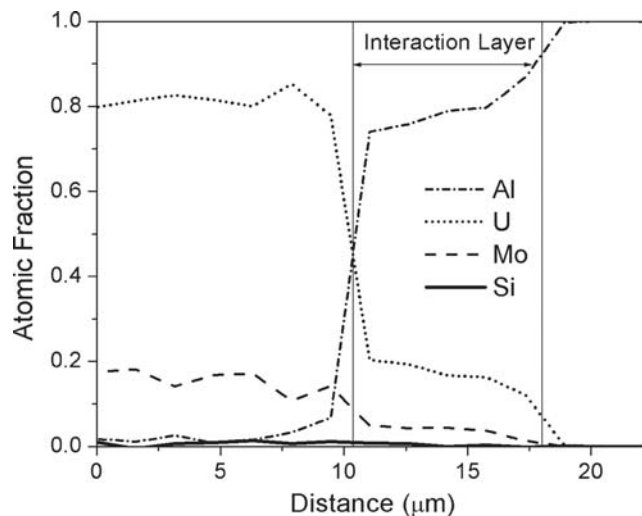


(b)

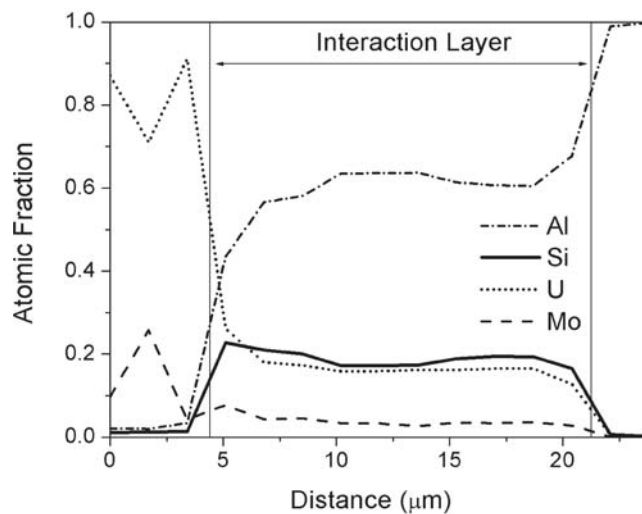
Fig. 7 (a) SEM micrograph of a U-7Mo/Al-2Si dispersion fuel sample annealed at 580 °C for 10 h. (b) Al/(U + Mo) atomic ratio profiles in interaction layers of U-7Mo-0.2Si/Al, U-7Mo-1Zr/Al, and U-7Mo/Al-2Si dispersion fuels annealed at 580 °C for 10 h

alloy was stable in the reaction phase, as shown in Fig. 6, suggesting that Si has a higher chemical affinity than Al for U.

The diffusion reaction behavior of the U-Mo-X/Al(-Si) dispersion fuel at 550 and 580 °C was observed. The dispersion fuels with various alloying modifications annealed at 580 °C for 10 h showed virtually identical reaction layer thickness, as represented in Fig. 7(a) for the U-7Mo/Al-2Si dispersion fuel. This implies that the effect of the alloying modifications used here and with the contents and temperatures studied is negligible as far as diffusion kinetics are concerned. The composition profiles of the reaction phase in the U-7Mo-0.2Si/Al, U-7Mo-1Zr/Al, and U-7Mo/Al-2Si dispersion fuels annealed at 580 °C for 10 h were plotted in Fig. 7(b). The U-7Mo-0.2Si/Al and U-7Mo-1Zr/Al dispersion fuels showed higher Al/(U + Mo) atomic ratio profiles in the reaction phase, whereas the U-7Mo/Al-2Si dispersion fuel showed a lower Al/(U + Mo) atomic ratio



(a)



(b)

Fig. 8 Compositional profiles in the interaction layer of U-7Mo/Al-2Si dispersion fuels annealed (a) at 580 °C for 10 h and (b) at 550 °C for 25 h

profile in the reaction phase. The Zr-to-U ratios in the U-7Mo-1Zr/Al dispersion fuel in the U-7Mo-1Zr and in the reaction phase remained unchanged, suggesting that Zr does not preferentially migrate to the reaction phase at this temperature.

Figure 8 shows the compositional profiles of the reaction phase in the U-7Mo/Al-2Si dispersion fuels that were annealed at 580 °C for 10 h and at 550 °C for 25 h. The latter showed higher Si content (up to 20 at.%) in the reaction phase, whereas Si accumulation in the reaction phase was not observed in tests conducted at 580 °C for 10 h. Because the minor change of Si content in Al is difficult to detect, the depletion of Si near the diffusion reaction phase was not measured in this study. Mirandou et al.^[20] observed the depletion of Si precipitates near the diffusion reaction phase in a U-Mo/4043Al diffusion couple test. The uphill diffusion of Si into the reaction phase has been predicted using

a semiempirical thermodynamic analysis performed by Kim et al.^[9] Considering that the diffusion couple test of U-Mo versus 6061 Al alloy at 340 °C showed 50 at.% Si composition in the reaction phase,^[21] the preferential accumulation of Si in the reaction phase of U-Mo/Al dispersion fuel is considered to be more substantial at lower temperatures.

4. Conclusions

The growth rate of reaction layers and the activation energy for the growth of reaction layers of U-Mo/Al dispersion fuels were obtained. The reaction phase of the dispersion fuel from high-temperature annealing tests and irradiation tests commonly showed a multilayered morphology with composition variation.

The γ stability of the U-Mo alloy was enhanced by the addition of a small amount (0.1 wt.%) of Si, whereas the effect of the addition of other elements such as Al was deleterious. The effect of alloying modifications on diffusion reaction kinetics from annealing tests at temperatures of ~550 °C for the contents up to ~2 wt.% was negligible. The annealing tests of U-Mo-Si/Al and U-Mo-Zr/Al dispersion fuels showed diffusion reaction behavior that was similar to a U-Mo/Al dispersion fuel.

Si, originally added into the Al matrix, was found to accumulate in the reaction phase of the U-Mo/Al-Si dispersion fuel annealed at ~550 °C, which is consistent with the thermodynamic estimation. However, the migration of Zr or Si to the reaction phase, when added to U-Mo, was not observed at this temperature.

Acknowledgments

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